

## GB1512549

Publication Title:

PROCESS FOR THE TREATMENT OF WOOD

Abstract:

Abstract of GB1512549

1512549 Preserving wood HAGER AB 26 Nov 1975 48660/75 Heading A5E [Also in Division D1] A process for treating wood comprises impregnating wood with a solution containing a preservative dissolved in a water-free organic solvent and thereafter treating the wood with a high boiling point oil composition at elevated temperature under vacuum, the difference in boiling point between the solvent and the oil being such that the solvent then evaporates from the wood. The impregnation step is preferably carried out in a metal cylinder using the Rueping or Lowry "empty cell" method terminating with a vacuum step. The preservative may be a chlorinated phenol or naphthalene, nitrophenol, cresol, naphthol, a fatty acid (6-12C) or a tin or copper compound e.g. tributyl tin oxide and is dissolved in the solvent optionally with the aid of a solubilising agent such as tall-oil or naphthenic acid. The solvent preferably has a boiling point below 200 C and may be a mineral oil such as toluene or xylene or a chlorinated hydrocarbon or a mixture of mineral oil with chlorinated hydrocarbon to provide fire retardancy. The high boiling point oil has a boiling point of at least 250 C and may be a drying oil such as linseed oil, tall-oil or an alkyd, or a non-drying oil e.g. a paraffin oil or a mixture of the two. It may contain pigments, fire retardants such as tricresyl phosphates and chloroparaffins, and also the preservatives mentioned above. The oil treatment step may be effected at between 60 and 110 C preferably at a pressure of 200 mm Hg or below, and provides a finish on the wood as well as the means for removing solvent. Data supplied from the esp@cenet database - Worldwide

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# PATENT SPECIFICATION

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## (54) PROCESS FOR THE TREATMENT OF WOOD

(71) We, HAGER AKTIEBOLAG, a Swedish Body Corporate, of Kryssavagen 17, 18352 Taby, Sweden, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the preservation of wood, and is concerned with the provision of a process whereby a wood preservative is administered to wood by the aid of a non-aqueous treatment solution.

United States Patent Specification No. 3,560,251 describes a process for treating wood comprising the impregnation of wood with an aqueous solution containing wood preserving agents followed by the treatment of the impregnated wood with a warm, high boiling point oil under vacuum. The impregnation step affords protection against wood destroying agencies such as decay and insects, and the oil treatment step effects fast, mild drying of the wood and affords a surface protection against moisture, cracking, etc. If the oil has been pigmented, the treated wood is colored. The process thus provides a fast, mild and comprehensive treatment for wood. The performance of the process is facilitated by the fact that the oil and the water (the preserving solution) are not miscible. In this way, the evaporation of the water from the wood is made easier.

Experiments have, however, made it clear that a similar process can, under certain conditions, be executed even if an organic solvent—soluble in the high boiling point oil—us used as diluent for the preservative. The conditions under which such a modified process can be effected will be hereinafter described.

In accordance with the present invention there is provided a process for treating wood, which process comprises impregnating wood in a treating vessel with a solution containing a preservative dissolved in an evaporable solvent removing the solution from the vessel and

thereafter introducing a treatment oil into the vessel, treating the wood with the oil under vacuum so that the solvent evaporates from the wood, and removing the oil from the treating vessel under maintained vacuum, wherein the evaporable solvent is an organic solvent having a boiling point of below 200°C and which is soluble in the oil, wherein the oil has a boiling point of at least 250°C and wherein the oil is maintained at a temperature of between 60° and 110°C throughout the treatment step.

The boiling points of the solvents and oils referred to herein are defined in each case at atmospheric pressure.

The process of the present invention requires that the wood be first impregnated with a preserving agent dissolved in an evaporable organic solvent. This impregnation step is carried out in any suitable treatment apparatus usually an iron cylinder. Thereafter, the solution is removed from the cylinder and the treatment oil is introduced. The oil is kept at between 60° and 110°C and, at the same time a sub-atmospheric pressure (a vacuum) is maintained within the treatment cylinder. Under these conditions, the solvent rapidly evaporates (boils) from the wood. The vapor from the solvent is condensed (cooled) and recovered for further use. The treatment oil whilst still being maintained under a vacuum is thereafter removed from the treatment cylinder, and the wood in the cylinder can be considered to be "ready treated". By means of the introduction of the preserving agent, the wood is provided with protection against destructive organisms and furthermore, it is given a surface protection against air and moisture by virtue of the oil which has been introduced into the outer layers of the wood.

The impregnation step is preferably carried out under pressure in an iron cylinder. In order to minimize the quantity of solvent evaporating from the wood during the following step, an "empty cell"

5 treatment method such as that described by  
Reuping or Lowry is preferred. In this  
method, Swedish pine, for example,  
receives from about 80 to 150 liters of  
solution per cubic meter of wood, instead  
of the 300 liters which would need to be  
employed if the "full cell" method of  
impregnation were to be used. Both of the  
named methods afford good distribution of  
10 the preservatives through the wood. The  
amount of preservative introduced into the  
wood is the same and independent of the  
method used. If the selected method uses  
less solution, the solution is kept stronger to  
15 compensate for the smaller amount of  
solution absorbed by the wood.

For preserving treatments based on  
organic solvents, the empty cell method can  
be looked upon as being an economic  
20 necessity. An expensive solvent is used, and  
it is a problem to keep the consumption of  
it as low as possible. When using  
conventional pressure treatment, the  
solvent cannot be recovered. After the  
25 treatment when the solvents have fulfilled  
their task by distributing the preservative  
they evaporate from the wood and in this  
way are lost.

It may be observed that when using the  
30 preferred process according to the present  
invention—wherein the Rueping and/or  
Lowry method is a part of the process—the  
reasons against the "full cell" alternative  
are not so strong as when conventional  
35 pressure treatments are used. By using the  
present process the solvent is recovered;  
the problem is to make this recovery as  
simple as possible. The "empty cell"  
methods tend to solve this problem and, as  
40 will be seen below, the cost of the recovery  
is not high.

In order to achieve optimum results using  
the present process, the solvent and the  
45 treatment oil in which the wood is heated  
must have certain properties which differ  
from one another: above all their boiling  
points or boiling intervals must possess  
special characteristics.

The organic solvent must be one which is  
50 evaporable and have a boiling point of  
below 200°C. A solvent with a very low  
boiling point however is preferably not used  
because of the risks relating to fire, health  
and other inconveniences, leakage, and  
55 other losses of the solvent, etc. Extremely  
evaporable solvents have been used in  
pressure treatment processes, but they have  
caused considerable difficulties. In order to  
obtain suitable operating conditions, the  
60 solvent is preferably in liquid form and does  
not have too high a vapour pressure at  
ordinary temperatures. If the solvents have  
boiling points which are less than about 125  
degrees Celcius they should desirably be  
65 composed of or contain substances which

are difficult to burn or have fire retardant  
properties. The boiling point must not on  
the other hand exceed 200°C., otherwise  
the evaporation of the solvent from the  
wood—carried out with the aid of the hot  
70 treatment oil—will be difficult to  
accomplish. Preferably, the boiling point is  
about 150°C.

As regards solvents, mineral oils having a  
limited boiling range are preferably used. 75  
Pure products derived from petroleum such  
as toluene and xylene may be used but  
they are more expensive. These pure  
products have the advantage of distilling  
(boiling) at an exact temperature; if they  
80 contain impurities they still distil within a  
very narrow temperature range. With these  
solvents it is easier to carry out the process  
with the best result and to recover the  
solvents most completely. 85

Solvents which are difficult to burn such  
as certain chlorinated products may be  
used with some advantage. These solvents  
are, for example, dichlorobenzene,  
90 dichlorobutane, trichloropropane,  
tetrachloroethylene, tetrachloroethane and  
pentachloroethane. When using solvents of  
this kind, lower boiling points can, as  
indicated above, be accepted; even such a  
low boiling product as trichloroethylene  
95 may be used. A preferred boiling point  
range is between 75 and 125°C. It seems  
best, however, to use a mineral oil and,  
when necessary, to mix it or the treatment  
oil with a fire-retardant chemical, which is  
100 not a preservative, such as a chlorinated  
organic compound or tricresylphosphate,  
generally in an amount of from 10 to 20 per  
cent weight based on the total weight of  
the solvent. As a fire retardant, a  
105 chlorinated naphthalene may, for example,  
be used.

A preserving agent such as, for example,  
penta-, tetra- or trichlorophenol,  
110 nitrophenol, cresol, or naphthol may be  
used, but tin compounds such as  
tributyltin oxide or organic oil-soluble  
copper-compounds may also be used. Fatty  
acids having from 6 to 12 carbon atoms  
such as caprylic and capric acid, and their  
115 copper compounds are also very suitable.

In some cases these preserving agents  
cannot be directly dissolved in the solvents.  
If so, the agents have to be dissolved or  
120 reacted with substances which make the  
agents soluble in the particular solvent that  
is used. Often tall-oil or naphthenic acid is  
used for dissolving copper compounds or  
chlorophenol.

Such a solution is introduced into the  
125 wood preferably by the Rueping or Lowry  
method. Both methods conclude with a  
vacuum step. It is preferable to maintain  
this vacuum when introducing the  
treatment oil into the cylinder. If the  
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vacuum is broken, it is desirable to let air enter the wood for a while. The introduced treatment oil can otherwise be forced into the evacuated wood to too great an extent by the air pressure. The treatment with the hot treatment oil tends to evaporate the solvent from the wood and to give a surface treatment; a deeper treatment is not required since it would consume too much of the expensive oil. The vacuum is also maintained because the following treatment step must be completed under vacuum.

The treatment oil must have a boiling point which is definitely higher than that of the solvent. The two liquids will probably be mixed with each other and if the boiling points are not sufficiently far apart a mixture of them will distill over, i.e. some azeotropic distillation will take place. This can be avoided—not theoretically but practically—if the boiling points of the two liquids are far enough apart. The boiling point of the treatment oil is therefore at least 250°C. But here also another point of view may be noted. A fraction of the treatment oil is desirably intended to remain in the surface of the wood to give permanent protection to the wood. During the useful life of the wood the treatment oil should preferably not evaporate. If the oil has to afford such protection it should preferably have a boiling point higher than 300°C.

Different kinds of treatment oils may be used depending on the circumstances. It can be a non-drying oil such as paraffin oil, or a less purified oil of this type, for example, a fatty oil. It can be a drying oil or a partly drying oil such as linseed oil, a tall-oil, or an alkyd. It can be also comprise a mixture of non-drying and drying oils.

If desired, a pigment can be added to the treatment oil in order to impart color to the treated wood. In addition other substances—for example, preservatives against blue stain and fire retardants such as tricresylphosphate and chloroparaffins can be added to the treatment oil. The preservatives normally present in the solvent can also be introduced into the treatment oil as well as the preservatives which are normally present in the treatment oil can be introduced into the solvent.

The treatment oil may be a single compound or a mixture of several compounds and is dependent on the end—use of the wood and on the substances added to the oil. As an example, it may be said that a pigment dispersion will be more stable if aromatic substances are present in the treatment oil. Drying oils may be present in the treatment oil mixture if a pigment is used. The drying degree of the drying oils may be limited if

the treatment oil is designed to allow the movements (shrinkage and swelling) of the wood without the surface of the wood cracking. As a rule a drying oil is present at least to some extent, but in the cases when only a surface protection against air and moisture is required a non-drying oil can be used exclusively.

When the treatment oil is introduced into the treatment cylinder preferably with the vacuum maintained, the treatment oil must be kept at a temperature of between 60 and 110°C. Heating can be effected in various conventional ways, e.g., by means of heating pipes within the cylinder, or by means of a mantle covering the lower part of the cylinder through which a heating medium such as steam or a hot liquid is passing. The heating temperature—the temperature of the oil—is preferably kept at about 80°C. The applied vacuum is preferably kept at a pressure of 200 mm Hg or below, more preferably about 100 mm Hg. At the beginning of this period a lower temperature and/or a lower vacuum may be used in order to retard the very fast evaporation of solvent during this part of the period.

When the solvent evaporates rapidly from the wood, it boils away owing to the presence of the vacuum. The wood will thus be “dried” free from solvent. Even if the same ideal distillation conditions do not prevail as when an aqueous solution is used in the impregnation step, experiments have shown that the results are acceptable (i.e. good) and definitely better than expected. This depends for the main part on the difference mentioned above between the boiling points of the solvent and the treatment oil. Also the solvent preferably has a lower heat of vaporization than the treatment oil: preferably it is at most only one-fifth that of water. This means a fast distillation; a low heat consumption; and cooling simply by the later condensation and recovery of the solvent.

It has been found that wood 25 mm thick can be dried almost free from solvent in about 4 hours, whilst for a thickness of 50 mm from 5 to 6 hours are needed.

The fact that the solvent and the treatment oil are mutually soluble and theoretically give poorer conditions for the distillation of solvent from the wood is thus compensated for in practice by proper selection of the solvent and the treatment oil, the preferred use of a solvent that has a relatively low heat of vaporization; and by suitable operating conditions.

The solvent can be distilled almost completely from the wood. During this distillation process some solvent will be dissolved in the treatment oil and the solvent is successively distilled away from

the treatment oil. It takes some time before the treatment oil is free from solvent—especially the last small amount thereof—and therefore, often the process is carried out with some of the solvent still left in the treatment oil. This amount is rather small and is constant—an equilibrium arises—if the process is effected in repeating cycles. With the boiling point intervals given above it is fairly easy to distil and recover treatment oil that is practically free from solvent at the end of the process, if this is desired.

As said before, the solvent is preferably recovered and this is best achieved by cooling. This is a simple process since the heat of vaporization of the preferred solvent is relatively low. Water or even air can be used as the cooling medium. The cooling stage is preferably placed between the treatment cylinder and the vacuum pump. In this way, the load on the pump will be low since it does not have to carry away any vapors.

The recovery of the solvent is, of course, very important when the solvent is expensive. The solvent can be used many times in its recovered state. Preferably no specific dissolving agents are lost. When carrying out the impregnation step with an aqueous solution—not of this invention—the situation is in this respect different. Here, the special dissolving agents for the preserving chemicals are not recovered. Such unrecovered dissolving agents can be ammonia, carbon dioxide or/and certain chromium compounds. Here we have a further advantage of the process according to the invention.

During the distillation period, the hot treatment oil will give the surface of the wood a thorough treatment. It may be noted that the treatment oil treats the wood for several hours at between 60° and 110°C; and that all parts of the treatment oil are available to the wood. The applied heat also gives the treatment oil a low viscosity during the treatment step. This can be contrasted to the use of a diluent for the oil which later evaporates and is lost. The treatment step is thus much more effective than ordinary painting, and, furthermore, it gives the wood surface at least 5 times as much treatment oil as painting does.

When the process has been effected as described it is terminated. This termination step will be carried out so that the treatment oil is pumped out of the cylinder and away from the wood. During this operation the selected vacuum has to be maintained otherwise the external air pressure would force too much expensive treatment oil into the wood.

As said before, any treatment oil remaining in the surface of the wood constitutes a protection of the wood against air and moisture. When pigment has been introduced into the treatment oil the wood will be coloured. It has been found that a net retention of treatment oil in the wood of about 40 kg per cubic meter of wood is obtained when wooden boards 25 mm thick are treated. Thinner wood, i.e. with a larger surface area, will absorb more treatment oil, and thicker wood less treatment oil, per cubic meter.

After the wood has been taken out of the cylinder it is "ready treated" and is ready for use. If drying oils have been used, several days storage of the wood may be needed to allow these oils to harden.

#### EXAMPLE

4.3 cubic meters of pine boards, each about 4 meters in length and with a dimension of 25×100 millimeters were treated.

The treating solution contained a mineral oil boiling between 150 and 165 degrees C. as the solvent. The preservative, pentachlorophenol, was dissolved in the solvent with the help of 3 per cent by weight of tall-oil.

The "empty cell" Lowry method was used for the treatment step. The preserving solution was accordingly introduced into the wood under a pressure of 8 kgs per square centimeter, for 90 minutes. Then the remaining solution was carried away, and a high vacuum was applied for 30 minutes during which period some of the preservation solution was driven out of the wood. By using the maintained vacuum, a treatment oil was introduced into the cylinder. It was composed of equal part of linseed oil and oil of the paraffin type having a boiling point of at least 250°C. In the oil mixture organic, microlite, red pigments (Ciba) had been dispersed.

The treatment step with the treatment oil commenced at 60 degrees C. After 60 minutes the temperature had been elevated to a final temperature of 80 degrees C. The period lasted a total of 4 hours and 15 minutes. The vacuum was held at about 100 mm Hg.

During this period a total of 650 liters of solvent evaporated. This solvent was condensed and recovered. After evaporation of the solvent, the vacuum was maintained and the treatment oil was pumped out of the cylinder. Finally, the treated wood was taken out of the cylinder and it was found to be practically free of solvent.

It may be noted that after 2 hours distillation an amount of about 4 per cent

solvent was estimated to be present in the treatment oil.

The treated wood had a red color and had been given surface protection provided by the treatment oil. After a couple of days the linseed oil had become hardened and the wood surface was "dry" and gave no "oil troubles" when the wood was handled.

#### WHAT WE CLAIM IS:—

1. A process for treating wood, which process comprises impregnating wood in a treating vessel with a solution containing a preservative dissolved in an evaporable solvent, removing the solution from the vessel and thereafter introducing a treatment oil into the vessel, treating the wood with the oil under vacuum so that the solvent evaporates from the wood, and removing the oil from the treating vessel under maintained vacuum, wherein the evaporable solvent is an organic solvent having a boiling point of below 200°C and which is soluble in the oil, wherein the oil has a boiling point of at least 250°C and wherein the oil is maintained at a temperature of between 60° and 110° throughout the treatment step.

2. A process as claimed in claim 1, wherein the impregnation step is carried out according to the Rueping or Lowry "empty cell" method.

3. A process as claimed in claim 1 or claim 2 wherein the treatment oil is maintained at a temperature of 80°C during the evaporation step.

4. A process as claimed in any one of the preceding claims, wherein the solvent has a boiling point of 150°C.

5. A process as claimed in any one of claims 1 to 3, wherein the boiling point of the solvent is between 75 and 125°C.

6. A process as claimed in any one of the preceding claims, wherein the solvent consists of a mineral oil.

7. A process as claimed in any one of the

preceding claims, wherein the solvent or the treatment oil contains a fire-retarding agent which is not a preservative.

8. A process as claimed in claim 7 wherein the fire-retarding agent is tricresylphosphate.

9. A process as claimed in claim 7 wherein the fire-retarding agent is a chlorinated organic compound.

10. A process as claimed in claim 9, wherein the chlorinated organic compound is a chlorinated naphthalene.

11. A process as claimed in any one of the preceding claims wherein the preservative is a poly-chlorophenol, a fatty acid having from 6 to 12 carbon atoms, a copper compound or a tin compound.

12. A process as claimed in any one of the preceding claims wherein the treatment oil has a boiling point which is higher than 300°C.

13. A process as claimed in any one of the preceding claims, wherein the treatment oil comprises a drying oil.

14. A process as claimed in any one of the preceding claims, wherein the treatment oil contains a finely dispersed colouring agent.

15. A process as claimed in any one of the preceding claims wherein the treatment step is performed at a pressure of not greater than 200 mm Hg.

16. A process as claimed in claim 15 wherein the treatment step is performed at a pressure 100 mm Hg.

17. A process as claimed in claim 1 substantially as hereinbefore described in the Example.

18. Wood when treated by a process as claimed in any one of the preceding claims.

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## GB1432649

Publication Title:

No title available

Abstract:

Abstract of GB1432649

1432649 Cinnoline derivatives IMPERIAL CHEMICAL INDUSTRIES Ltd 23 July 1974 [7 Sept 1973] 42173/73 Heading C2C [Also in Division A5] The invention comprises compounds of the Formula I where R<SP>1</SP> is H or C 1 to C 6 alkyl; R<SP>2</SP> is H, C 1 to C 5 alkyl, C 3 to C 5 alkenyl, or benzyl, and R<SP>3</SP> is H, C 1 to C 5 alkyl, C 1 to C 5 alkoxy, phenyl, benzyloxy or methylenedioxy; and non-toxic pharmaceutically-acceptable salts thereof. Compounds of Formula I in which R<SP>1</SP> and R<SP>2</SP> are H, may be prepared by [reacting a benzazocine derivative of Formula II with an alkyl nitrite and an inorganic acid in the presence of a small amount of water. Compounds of Formula I in which R<SP>1</SP> is C 1 to C 6 alkyl and R<SP>2</SP> is H may be prepared by esterifying the corresponding carboxylic acid in which R<SP>1</SP> is H, or a corresponding acid halide or anhydride. Compounds of Formula I in which R<SP>1</SP> is C 1 to C 6 alkyl, and R<SP>2</SP> is C 1 to C 5 alkyl, C 3 to C 5 alkenyl or benzyl may be prepared by reacting an alkali metal derivative of the formula where M is Na or K, with a halide reactant of formula R<SP>2</SP>Hal where Hal is Cl, Br or I. Compounds of Formula I where R<SP>1</SP> is H may be obtained by hydrolysing in the presence of water a compound of formula where Cy is alkoxycarboxyl, phenylalkoxycarbonyl, phenoxycarboxyl, cyano, carbamoyl or thiocarbamoyl. The benzazocine starting materials of Formula II may be prepared by heating a phenylhydrazine derivative of formula with cyclopentanone to produce an indole derivative of formula followed by treatment of the latter with sodium periodate in methanol/water to form the benzazocine derivative II. Data supplied from the esp@cenet database - Worldwide

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